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Grain boundary structure and mechanical properties

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Résumé.— Une description détaillée de la structure des joints de grains est faite, en se concentrant sur les modèles qui peuvent être vérifiés sur la base des résultats expérimentaux dans les polycristaux. Une revue est également faite des études expérimentales récentes consacrées aux aspects cristallographiques en liaison avec la texture de désorientation des grains. On montre que le lien est fort entre les propriétés et les caractères géométriques des joints de grains. Ce lien est particulièrement important dans le cas des propriétés mécaniques.

Abstract.— A comprehensive review of grain boundary structure is given concentrating on models which can be tested on the basis of experimental results from polycrystals. A survey is also given of recent experimental studies, centered upon the crystallographic aspects in terms of the grain misorientation texture. It is shown that there is a strong connection between properties and the geometrical character of a grain boundary. This connection is particularly important in determining mechanical properties.

1. Introduction

The strategy of approach to the relationship between grain boundary structure and properties, with particular reference to mechanical properties, begins by reviewing the models which define the geometrical parameters of the boundary. Secondly, the present understanding of how boundary geometry influences mechanical and other properties is outlined. The next section illustrates some of these structure/property relationships by reference to examples from the authors' recent experimental work. The emphasis is then turned toward the mechanical behaviour of polycrystals in the light of structural influences. Also, the philosophy of "grain boundary engineering" is discussed as a goal, which can be achieved through developing and extending knowledge of boundary structure/property effects.

2. Grain boundary structure

In order to describe the geometrical relationship between any two grains, it is necessary to define five main degrees of freedom. Two degrees of freedom define

the axis of misorientation, ℓ , and one is required to specify the angle of misorientation, θ . If the first grain is notionally rotated through θ about ℓ , by definition the orientation of the second grain is achieved (figure 1). The remaining two degrees of freedom are required to define the orientation of the boundary plane. Any experimental analysis of grain boundary structure is based on measurement of at least the first three of these parameters.

Once the axis/angle pair, ℓ/θ has been determined, the first division of grain boundary structure is apparent, that is, whether a boundary falls into the high angle or low angle ($\theta < 10-15^\circ$) class. The structure of low angle boundaries is characterised by arrays of lattice dislocations, termed primary intrinsic grain boundary dislocations, which totally account for the misorientation and the relaxed structure [1]. This type of boundary configuration is well understood. At misorientations between $10-15^\circ$, the overlap between neighbouring dislocation cores means that the individual identity of primary dislocations is lost and the boundary can no longer be characterised in

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such simple terms. The largest value of θ for which primary dislocations have been recorded in a boundary is 15° [1]. For larger misorientations $\theta > 10-15^\circ$, the traditional view is that many boundaries are randomly oriented with respect to their neighbours, but some are characterised by non-random geometrical relationships. One of the first expressions of this type of structured boundary is due to Mott[2], who developed the transition lattice model of Hargreaves and Hill to describe boundaries as regions of good fit interspersed with regions of poor fit. From this simple approach many structural theories subsequently evolved; they all feature the common theme that, for a proportion of boundaries, there is a repeating arrangement of the adjoining atoms at the boundary. The concept of periodicity has been quantified by several approaches, but only the coincident site lattice (CSL)[3] and coincident axial direction/planar matching (CAD/PM) models [4,5] will be described in detail here since they are the most easily applicable models to large statistical studies of many boundaries in polycrystals. For certain combinations of l/θ , some lattice points of grain will coincide with those of grain 1 to give a superstructure of coinciding sites[3]. Figure 2 shows a 36.9° rotation around $\langle 100 \rangle$ which causes 1 in 5 lattice points from grains 1 and 2 to coincide; this is referred to as a $\Sigma = 5$ CSL. At the boundary plane, the periodicity is on average $5^{1/3}$ since the volume of the CSL cell varies as Σ [6]. The ordered arrangement of atoms in a CSL boundary often allows much closer packing in the grain boundary plane than for the disordered case, particularly where rigid body translations permit the boundary to relax to a better fit[7].

A CSL exists only for precise values of l/θ . However, the CSL structure can be conserved by arrays of dislocations which maintain the 3-dimensional superstructure. Such dislocations are termed secondary intrinsic grain boundary dislocations since they take up the angular deviation from an exact high density (low Σ) CSL in a manner precisely analogous to the misorientation across a low angle ($\Sigma = 1$) boundary being maintained by primary grain boundary dislocations. There is much TEM evidence for the existence of near CSL boundaries in the form of arrays of grain boundary dislocations (e.g 6). Not all the evidence is the result of oriented bicrystal studies: structured boundaries are known to also exist in polycrystals (e.g. 8, 9).

If high enough values of Σ are allowed, any boundary may be described in terms of a CSL, although the physical significance of very long period boundaries is probably small. For values of Σ greater than about 49, it is more convenient to consider matching in one dimension only rather than three, because at this stage the three

Burgers vectors of grain boundary dislocations, b_1 , b_2 , b_3 , have become extremely anisotropic with the largest Burgers vector, b_3 , approaching the dimensions of the interplanar spacing while b_1 and b_2 become extremely small[10]. This type of one dimensional matching is described by the PM theory[5], or if considered from the standpoint of the deviation from parallelism of the plane stack normals in the direction of b_3 , the CAD theory[4]. Support for the PM theory comes from the observation of dislocations in boundaries which are several degrees away from the nearest low index CSL (e.g.11) and, as will be discussed in a later section, the PM/CAD approach is particularly suitable for application to textured materials (e.g. 12).

Other approaches to grain boundary geometry have contributed to the present understanding of boundary structure. Bollman's 'O' lattice model enables a completely quantitative description of any boundary to be formulated. It is a continuous function of grain misorientation and so CSL's emerge as a special case where the 'O' points - regions of best match - coincide with lattice points[13]. Formulation of the DSC lattice (a sublattice of the 'O' lattice) allows possible Burgers vectors of grain boundary dislocations to be predicted. The structural unit model[14] also arose from the fundamental premise of fit/misfit regions in the boundary, but adopts the view that poor fit regions are equivalent to the insertion of small groups of atoms, which give rise to ledges at the boundary. Those ideas later evolved into the "random close packed units" model which considers that various polyhedra of atom groups provide the basic building blocks of boundary structure[15]. Finally, the current knowledge of boundary structure could not have progressed to its present level without the recent atomistic studies of boundary geometry made possible by the advance of high speed computers (e.g. 16).

2.1 Proportions of special boundaries in a sample

In order to calculate the proportion of special boundaries that could result from a random distribution of misorientations, a knowledge of the maximum deviation from the exact CSL, V_{m2} (or CAD V_{m1}) which can be accommodated by grain boundary dislocations is required. Most workers choose the CSL limit as [3]

$$V_{m2} = V_0 \Sigma^{-\frac{1}{2}} \quad (1)$$

where V_0 is the angular limit for $\Sigma = 1$, i.e. 15° . Similarly the CAD limit is defined as [4]

$$V_{m1} = (a/b) \pi^{-\frac{1}{2}} \quad (2)$$

where a is the lattice parameter, b is the

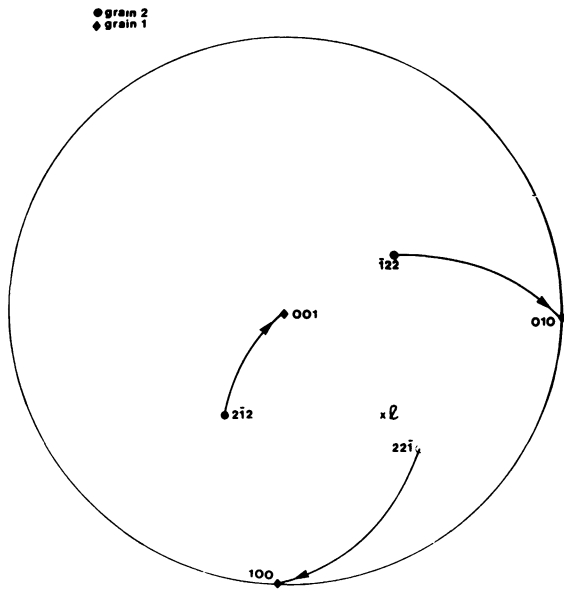


Figure 1. Stereographic representation of a twin-related bicrystal (illustrating the definition of ℓ/θ (see text)). A clockwise rotation of 60° (θ) about the common $[111]$ axis 1 rotates the $[212]$, $[\bar{1}22]$ and $[22\bar{1}]$ directions in crystal 2 on to the crystal 1 axis, $\langle 100 \rangle$ of crystal 1 (Courtesy J. Mat. Sci. reference [23]).

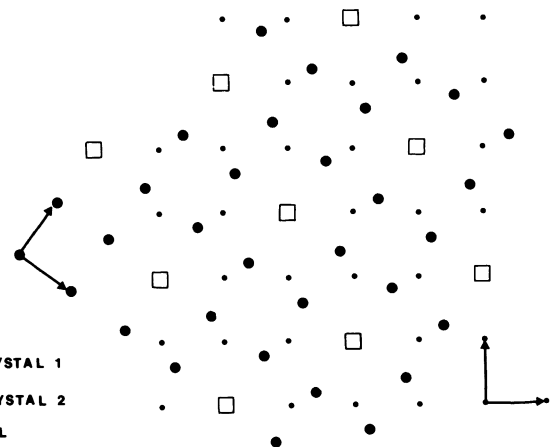


Figure 2. Illustration of the geometrical relationships giving rise to a $\Sigma = 5$ coincidence site lattice (CSL).

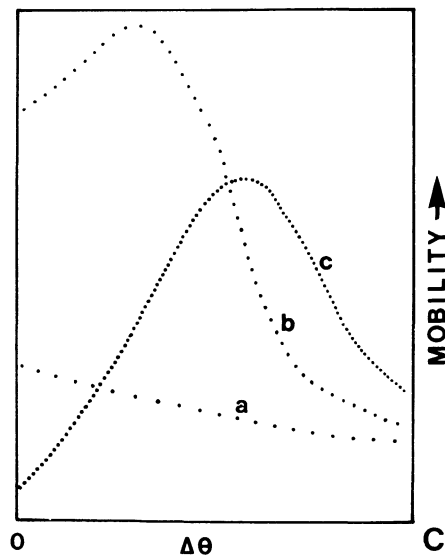
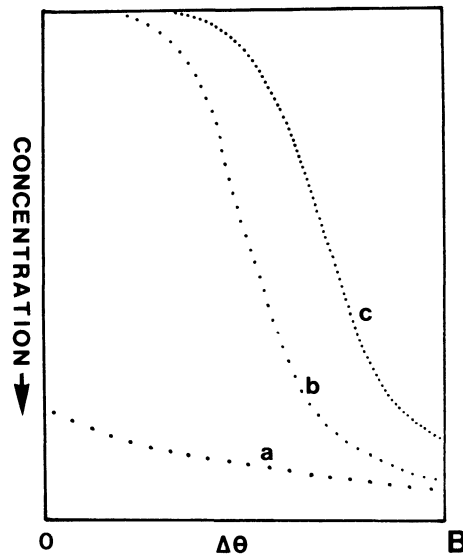
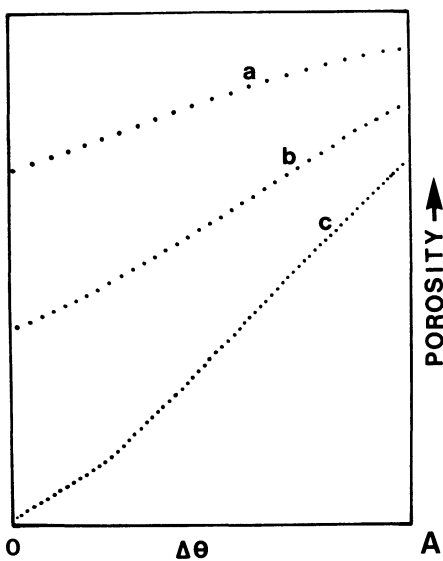


Figure 3. Schematic plots of (A) porosity functions (B) concentration function of segregant and (C), the sum of A and B, boundary mobility as a function of misorientation. $\Delta\theta$. a, b and c refer respectively to random, medium density and high density CSL boundaries (After Gordon and Vandermeer, [21]).

Burgers vector and $\pi = h^2 + k^2 + l^2$ for axis of misorientation hkl . These limits are not absolute and intended as guidelines only, particularly since the limits to experimental resolution prevent the detection of very closely spaced dislocation arrays. It is suggested by some workers that, as further dislocation hierarchies are "pressed into service", larger deviations than those suggested by equation (1) may be represented by a dislocation model [17]. In fact, it is quite possible that most if not all grain boundaries may be located in an "energy valley" [4]. It has been shown that if a completely random distribution of rotations is generated, for cubic materials 9% of these rotations should be special boundaries with $3 < \Sigma < 25$ and 2% low angle types [4]. For the CAD case, it is estimated that about 50% of all boundaries should have axes lying within an angular range within V_{m1} of the normals to $\{200\}$, $\{220\}$ and $\{111\}$ plane stacks. Any deviations from a random distribution of grains would be expected to change these proportions.

2.2 Boundary structure/segregation effects

The segregation to grain boundaries of elements which have limited solid solubility in the host matrix is frequently accompanied by changes in the boundary's energy and mobility. Even materials which are nominally pure may show orders of magnitude change in boundary migration rate due to the presence of less than 0.003% solute [18]. A few segregants in specific systems such as B in Ni are "cohesive enhancers" but the majority of segregating species such as S and P in Fe are the cause of embrittlement in grain boundaries [19]. However, from a number of investigations involving both bicrystals and polycrystals (e.g. 20) it is now clear that boundary segregation is extremely orientation dependent and that special boundaries (CSL and low angle) are far less susceptible to segregation than are random boundaries. The key to this behaviour is that, since special boundaries are relatively close packed compared to random geometries, there is less free volume associated with special boundaries and consequently less available space for segregating species. The relationship between boundary porosity, mobility and local segregant concentration has been explored by Gordon and Vandermeer, who postulate that boundary mobility is a sensitive function of both porosity and segregation [21]. If curves schematically representing the relationship between both boundary porosity and local solute concentration with misorientation from exact matching are summed, the boundary's mobility may be predicted [21]. If random, high density CSLs (e.g. $\Sigma = 3$) and lower density CSLs are treated separately, it becomes clear from the schematic plot of mobility versus orientation function in figure 3 that the

mobility of CSL boundaries is higher than that for random boundaries for all values of θ except high density CSLs at or very near exact coincidence. The maximum mobility occurs for medium coincidence boundaries, which deviate from precise coincidence by a few degrees. High density CSLs at exact coincidence (the coherent twin is a well known example) tend to be almost immobile because the atomic packing is so close as to resemble the lattice and consequently the activation energy for migration is less than for a disordered boundary, where its associated free volume contributes to a low activation energy.

Where a system is supersaturated with respect to grain boundary solutes, it is therefore likely that random boundaries will predominate over CSLs. These concepts which relate segregation effects to boundary structure can be extended to include the situation where supersaturation at the boundary is relieved by precipitation [22]. After precipitation, less free volume is taken up at the boundary by solute, and therefore a high proportion of special boundaries should be favoured than for the boundary-supersaturated regime.

The models of boundary structure outlined in the previous sections, and the way in which they relate to certain boundary properties, will now be discussed in the light of recent experimental results by the present authors.

3. Evidence supporting theories of grain boundary structure/property relationships

The first step towards relating experimentally acquired knowledge of boundary structure to boundary properties and behaviour is the collection of a large enough data base to be able to draw statistically based conclusions concerning structure/property control. The present authors have used experimental techniques based either upon convergent beam electron diffraction CBED in a TEM [23], or electron back scattering EBS in an SEM [24] to collect data. The relative merits of both approaches are listed in table 1. In the remainder of this section, some aspects of these investigations will be summarised.

Although the CAD model can be treated with the same formalism as the CSL model, the former has not previously been applied to the analysis of boundary structure. The present authors have collated axis/angle pairs from materials of increasing complexity - pure copper, a two phase alloy of Al and Al₂O₃, a single phase austenitic stainless steel, a ferritic stainless steel and a complex nickel based superalloy - and categorised them according to the nearest CAD axis [25]. A comparison is then possible between the theoretical and observed

proportions (table 2). The distribution of axes varies from the predicted frequencies, which the authors describe as "grain misorientations texture", GMT. The existence of these textures could be either the cause or the result of grain texture[6]. In other words either a preferred orientation may exist in the material which directly controls the GMT or misorientations are selected by differentials in migration rates, energies or chemistry. The preferred orientation case applies to the Al/Al₂O₃ alloy which has been drawn to a wire and contains a high proportion of <200> CAD's (table 2). The other two subgroups in table 2 which exceed the theoretical frequency - <111> of the austenitic stainless steel and <200> of the ferritic stainless steel - are not strongly textured. Where a material is weakly or multi-textured, the investigations which have been conducted so far indicate that the effect on the GMT is very complex. For example, figure 4 illustrates a large secondary recrystallised grain in a complex superalloy, the distribution of grain normals of the large grain and surrounding small grains, the GMT of boundaries between small grains, and the GMT of boundaries between the large grain and surrounding small grains. On such GMT diagrams as figure 4c and d it is only possible to represent λ/θ and not λ/θ , but special combinations of λ/θ , i.e. CSLs, are indicated. It is evident that the grain growth process has changed the GMT from CSL's having near <220> (small/small grain boundaries) to CSLs with near <111> (large/small grain boundaries). Since the orientation of the large grain is not differentiated from the surrounding small grains, as illustrated by the distribution of grain normals in figures 4b, in this case the GMT is not the result of preferred orientation.

The theories concerning effects of segregation on grain misorientations which are outlined in the previous section, are further supported by results from a nickel-based superalloy, Nimonic PE16, after various heat treatment conditions[8]. The mechanical properties of this material are dependent upon precipitation of M₂₃C₆ (based on Cr₂₃C₆) and γ', (based on Ni₃(Ti,Al)). When the alloy is unaged with respect to both these phases, considerable solute supersaturation results.

Table 3 shows the proportions of CSL's present (a) in the supersaturated state (b) after an ageing treatment has allowed both Cr₂₃C₆ and Ni₃(Ti,Al) to precipitate thus removing a large proportion of segregant, (c) following a 1000h anneal which permits considerable coarsening, and (d) following re-resolution of γ' coarsened during (c). Over three hundred boundaries were sampled altogether. There is a clear trend for a progressive increase in the proportion of CSL boundaries and decrease

in the preference for random boundaries as precipitation increases, and boundary segregation decreases. When the overaged specimen is reannealed and quenched to retain the solute supersaturation caused by solutionisation of the γ'-forming elements, there is an accompanying return to a preference for random boundaries. For all of the heat treatment conditions the CSLs, while within the special boundary criterion given in equation (1), are mostly $>0.5 V/V_m$ where V is the measured angular deviation of a sampled boundary from an exact CSL. The implication is that these boundaries, which are a few degrees removed from exact CSLs, are more mobile than the random boundaries in the sample population, and that the former have been selected during growth on a structural basis. The large proportion of CSL's after prolonged ageing, and particularly the increase in nearer-ideal CSLs for this subgroup from 5% to 16%, (table 3) is only partially justified on the grounds of reduced segregation, since once precipitate growth is completed and true coarsening begins, the proportion of segregant at boundaries will be stabilised[26]. The dominant mechanism for the increased incidence of CSLs in this case is considered to be boundary sliding and is described in a later section.

The relationship between the incidence of CSL boundaries and intergranular precipitation is currently being further explored in a 310 stainless steel with M₂₃C₆ precipitation at grain boundaries. The EBS technique is being employed to collect crystallographic data from many boundaries. Figure 5a shows the variations in precipitate density over a typical region, and also indicates which of these boundaries are low angle, random or CSL (those boundaries which are obviously twins were omitted from the sample). The low angle and CSL boundaries commonly show low or medium density of precipitates and the random boundaries are characterised by medium or high density precipitation. For the whole sample population, only 14% of boundaries do not fit these categories (Table 4). Furthermore, the percentage of CSL's before precipitation of M₂₃C₆ have increased from 26.5% to an after precipitation value of 34%, which is consistent with the previous results from the superalloy. There are indications on Figure 5a that the boundary plane orientation also is significant in the control of the precipitate density, and investigation of this parameter and its influence on precipitate density is planned.

4. Mechanical Properties and the Influence of Grain Boundary Structure.

Grain boundaries have a major effect on most of the physical and chemical properties of materials. Nowhere is this

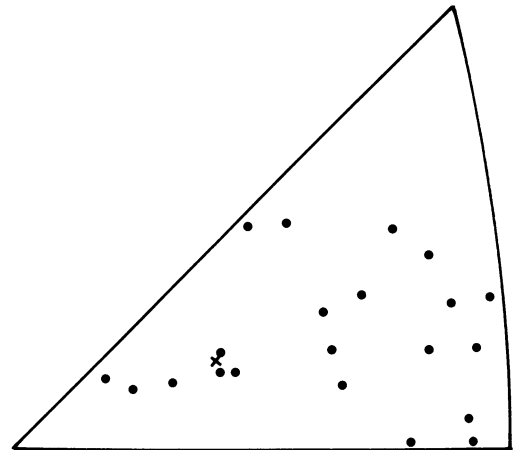


Figure 4. a) Scanning electron micrograph from Niimonic PE16 specimen showing the coexistence of large and small grains due to anomalous grain growth.

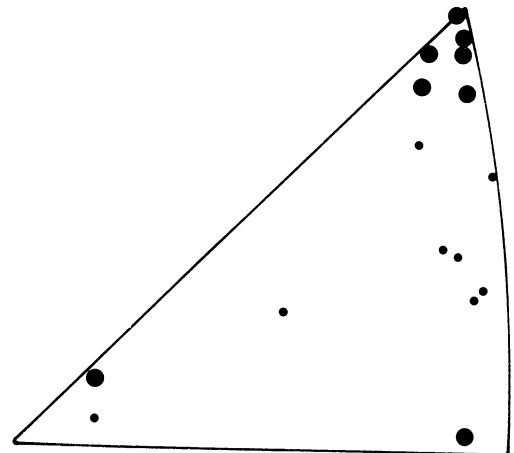
b) Stereographic unit triangle showing the orientation of the grain normals for a large grain (x) and the surrounding small grains (.).

c) Grain misorientation texture (GMT) plot of the small grains in figure 4a) with the larger symbol referring to CSL situations.

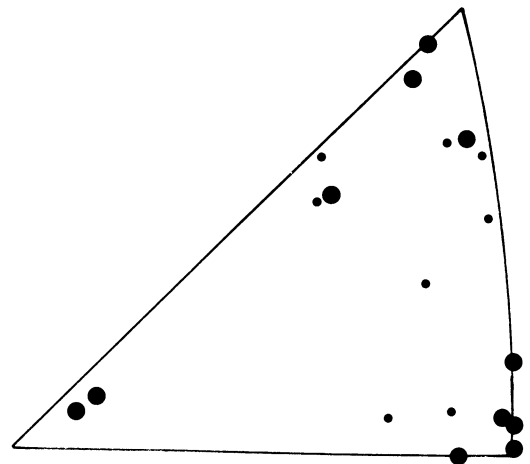
d) GMT as c) but for the boundaries between the large grain and surrounding small grains in figure 4a).



b



c



d



Figure 5 Scanning electron micrograph from sample of type 310 austenitic stainless steel showing variations in the density of $M_{23}C_6$ precipitates at boundaries. Boundaries with special geometries are marked S whilst those of random character are marked R.

TABLE 1

Comparison of the EBS/SEM method of grain boundary structural analysis (24) with the CBED/TEM method (23).

EBS	CBED
Accuracy for measurement of λ/θ 1-2°	Accuracy for measurement of λ/θ 0.5°
Data analysis on-line and semi-automatic, therefore very rapid.	Data handling under operator control from diffraction patterns therefore slower.
Many hundreds of grain boundaries available in a single sample.	Typically 20-30 boundaries available in a in a single foil.
The grain size distribution can be analysed in terms of grain micro-texture and related to the GMT.	No grain size specific information.
Information available restricted to that pertaining to grain orientations.	Grain boundary planes, dislocation characters and atomic level composition profiles can be determined.

TABLE 2

Proportions of CAD boundaries % in five different materials.

Material	$\langle 111 \rangle$ (28)*	$\langle 200 \rangle$ (12)*	$\langle 220 \rangle$ (16)*	total $\pi < 24$ (73)*
Cu	24	2	15	63
Austenitic steel	33	7	9	70
Al/Al ₂ O ₃	6	24	9	76
Ferritic steel	12	12	20	70
Nimonic PE16	23	7	12	63

*Theoretically derived CAD proportions (%)

TABLE 3

Proportions of CSL's (%) in Nimonic PE 16 as a function of ageing heat treatments. Both the special boundary criterion from equation (1) and an angular range equal to half this criterion, are used to classify the boundaries.

Heat treatment	$V/V_{m2} < 1$	$V/V_{m2} < 0.5$
unaged	10	5
aged	21	5
overaged	40	16
solution treated after overageing.	21	5

TABLE 4

Relationship between grain boundary precipitation of M₂₃C₆ and boundary "specialness" in 310 stainless steel (see also figure 5).

Number of boundaries	% special boundaries (near CSL $3 < \Sigma < 43$ or low angle)	% special boundaries having L-M precipitation	% random boundaries having M-H precipitation
137	44	84.5	77

L = zero or low density

M = medium

H = high density

more in evidence than in the response of a polycrystal to stress. The interest then in grain boundary/mechanical property interrelations spreads from the microscopic to the macroscopic; although it is the former which most concerns us here. The more macroscopic influence is best expressed in terms of equations of the Hall-Petch type which demonstrate an increase in strength and toughness with decrease in grain size. At the simplest level the strength improvement is attributed to the ability of a grain boundary to limit the motion of dislocations on slip planes by acting as a barrier or obstacle. In reality, this gives a simplistic picture of the interaction between dislocations and grain boundaries which needs considerable refinement (see below).

The influence of grain boundaries on the fracture mechanism and hence toughness is again extremely complex; only now are the type of grain boundary structural ideas referred to in previous sections beginning to be incorporated into models of the fracture of polycrystals. Here considering the ductile/brittle behaviour involves first looking at interactions between particular grain boundaries having specific structures and any solute impurities which may differentially segregate to these boundaries depending on their structure. This may then create an easy fracture path along those boundaries where the separation energy, which incorporates the grain boundary energy in the presence of these solutes, is low. Other factors come into this approach which have to consider other fracture paths through the grains and the local plastic work, if any, associated with the fracture process.

In general then, for combinations of high strength and toughness with good fatigue resistance, a fine grain structure is known to be optimum. It may well be possible to improve these properties still further by controlling the relative proportion of special (and therefore low segregation, low energy) boundaries (e.g. 20). This takes the subject of texture control (or tailoring) of polycrystals a step further in suggesting that it is not just the overall texture (or the orientation distribution function) which is important but that the local texture, GMT, (or grain boundary parameters) need to be controlled to achieve further improvements to properties.

The "design" of materials for operation under load at high temperatures brings into consideration different properties of grain boundaries. Here, under creep conditions we are mainly concerned with the ability of the grain boundary to resist sliding and to avoid the accretion of voids from vacancy condensation. At the simplest level we can minimise or remove these effects by going to specific

grain geometries (for instance in directionally solidified solids with the grain boundaries parallel to the stress axis) or by using single crystals. An alternative approach is to "design" the microstructure in the grain boundary regions; for instance by the incorporation of grain boundary precipitates which help to limit sliding. In the future we might hope to modify the microtexture, GMT, so as to maximise the percentage of grain boundaries within the polycrystal which have low sliding and vacancy capture characteristics.

Whilst not directly within the remit of this survey it is perhaps worthwhile stressing that a grain boundary not only has its own geometrical structure but is usually, in a practical alloy, associated with its own local microstructure. In general, a grain boundary represents a site where phase transformations are favoured and this in turn frequently leads to a region near to the grain boundary where there is some depletion in phase transformation products (e.g. precipitate free zone formation). This modified pattern in the microstructures at and near to the grain boundary may lead to very substantial changes in properties. It should be appreciated that this "grain boundary microstructure" is another manifestation of the grain boundary structure which may then have an even more dominant effect on the properties of a polycrystalline sample of a commercial alloy.

Many of the ideas expressed above are beginning to be considered within the remit of the materials engineer so that designing for instance for strength includes looking to increase the percentage of special boundaries[20]. This design process is at an early stage and is related in a number of papers in these proceedings and in the recent literature. In the present paper we limit our discussion to looking at some of the more microscopical/geometrical effects on low temperature (next subsection) and high temperature (following subsection) mechanical properties.

4.1 Low Temperature Mechanical Properties

Grain boundaries contribute to strength and toughness in a manner which is reflected in the overall properties of polycrystals. Further, the strength of single boundaries in bicrystals has been measured and shown to vary with the geometrical parameters which define the grain boundary. As yet, a full explanation for these hardening effects is not available although many of the contributions which grain boundaries make to the strength of polycrystals is becoming clearer.

It is believed that grain boundaries are the main sources of dislocations in

polycrystalline alloys. Further there is extensive evidence from analytical electron microscopy of the way grain boundaries act as barriers for dislocation motion creating pile ups. Examples of pile ups at grain boundaries are given in figure 6. Figure 6a shows an example of a planar array of dislocations crossing a grain and hence transferring stress/strain across the grain. This is typical of the form of behaviour seen in polycrystals of an alloy at low strain, although in this case the dislocations are in pairs due to the presence of a fine dispersion of the ordered intermetallic precipitate (γ'). Observations of this type, which are common, give rise to two questions which are strongly interrelated:

a) how do the dislocations which are forced into the grain boundary plane modify the grain boundary structure;

b) how does the process of strain transfer from grain-to-grain occur?

The answer to the second question is of considerably more practical importance since, in the absence of this strain transfer, fracture will occur. Whilst a full model for the strain transfer mechanism has yet to be developed, many of the key elements in such a model are now clear. Thus a single slip system in one grain intersecting a grain boundary will, in general because of geometrical constraints, cause more than one slip system in the neighbouring grain to be activated. Such considerations lead to a physical interpretation of the number of independent slip systems needed. At a more "microscopic level" this compatibility of operating slip systems may be interpreted in terms of densities of geometrically necessary dislocations [27]. Recent treatments of both the yield and the flow stress have started from this type of approach.

At a still more microscopic level are observations of individual dislocations reacting with the intrinsic structure of grain boundaries of which figures 6b and 6c are typical examples. In both these cases the periodic intrinsic structure of the grain boundary is resolved and the perturbation to this structure is apparent when a matrix dislocation enters into it. The processes by which this accommodation of matrix dislocations into the grain boundary structure takes place has been the subject of a number of studies although only a partial picture, as yet, emerges. There is also a suggestion that this accommodation process is different in a special boundary (possessing a short period structure) and a more general high angle boundary. In the former case, microscopic evidence exists which supports the idea that the matrix dislocation dissociates in the grain boundary plane to give secondary extrinsic grain boundary dislocations with the same Burgers vectors

as the intrinsics. The line vectors of these dislocations are initially governed by the zone axis of intersection of slip and boundary planes. Because these extrinsic dislocations perturb the periodic structure they have long range strain fields (and hence give stronger diffraction contrast than periodic intrinsics with the same Burgers vectors), and have a dominant effect on properties. Given combinations of glide and climb of these extrinsics in the grain boundary plane, they may eventually accommodate to become part of the periodic intrinsic structure. There is less certainty as to what happens when a dislocation enters a random high angle boundary but again one or more extrinsic grain boundary dislocations are formed although the cores of these may subsequently spread so that the dislocations are eliminated.

As deformation proceeds the dislocation substructure builds up from the grain boundary into the matrix. In many metallic materials this is seen by the formation of even more regular cells which begin at the grain boundaries and spread back into the grains. Figure 7 illustrates this phase of the deformation process in two different metallic specimens. In general at moderate strain levels (around 0.10) the cell structure near the grain boundary is smaller than it is in the centre of the grains reflecting the increased dislocation activity which arises from the additional geometrically necessary dislocations due to the requirements of strain compatibility across the grain boundary.

4.2 Grain boundary sliding

Grain boundary sliding is considered to be significant at temperatures above $0.4 T_m$, and leads to grain boundary diffusion - related phenomena such as creep and cavitation. However, where the sliding is not stress - related, a different sliding phenomenon has been observed. Usually, during a post recrystallisation anneal an alloy system will attempt to lower its energy via a reduction in grain boundary area - i.e. grain growth. However, the situation arises for Nimonic PE16 containing coarsened γ' that boundary migration is totally suppressed by the strong interaction of boundaries with γ' precipitate population. This strong interaction is a consequence of the maintained coherency of γ' during coarsening, and is described in detail elsewhere[8]. Although the translational motion of boundaries is restricted, they are still relatively free to slide or rotate about their axes. In so doing, it is possible for more energetically favourable configurations to be achieved and thus, for a pinned microstructure, the lack of grain growth associated energy reduction circumvented. It has been proposed that the large increase in the



Figure 6. Transmission electron micrographs of dislocation pile ups in Nimonic PE 16 a) shows a planar array crossing a grain b) shows a pile up entering a near $\Sigma = 9$ boundary and c) shows an individual dislocation entering a low angle grain boundary.

proportion of CSL's in PE16 containing strongly pinned, immobile boundaries (table 3) is a direct consequence of grain rotations by grain boundary microsliding [8]. Although the precise nature of the relationship between Σ value and grain boundary energy is complex and still not well understood, [28] it is generally accepted that CSL's with fairly low Σ values have lower energies than random boundaries. Hence the hypothesis that CSLs are energetically preferred configurations which boundaries will attempt to approach through sliding, is reasonable.

The sliding mechanism is a well accepted process during creep deformation, and the mechanism of sliding discussed here may be partially analogous to the creep case. Evidence from bicrystals exists for grain boundary rotations towards a CSL by the assimilation of lattice dislocations into the boundary [29]. Additionally, the overall sliding to new boundary orientations necessarily involves cooperative behaviour between boundaries in order to allow grain boundary dislocations to slide from boundary to boundary with suitable Burgers vector adjustments at grain corners. Evidence for the occurrence of adjustments to GMTs which involve complex interactions between grains is not just restricted to the case where boundaries have been strongly pinned for very long times at elevated temperatures, as cited here for PE16. Other systems, including 310 stainless steel discussed in an earlier section, may show clusters of CSL or near CSL boundaries (e.g. figure 5). The implication is that once one or two boundaries which border the same grain have rotated into CSLs (or were already CSLs as a result of grain texture or random selection) it is easier for other local boundaries to also become more ordered.

5. Concluding remarks

Essentially the theme of this article has been to show that a strong link exists between mechanical behaviour and grain boundary structure because of the property variations associated with special/non special boundary classifications. Generally we can say that a high proportion of special or ordered boundaries are beneficial to both strength and ductility, and clearly the ultimate aim is to design materials in such a way as to optimise their grain boundary properties. Before "grain boundary engineering" can be usefully implemented, there is an urgent need to extend our understanding of the factors which influence grain misorientation distributions. The authors have indicated some of their areas of activity in this important field, and their philosophy is the statistical study of many boundaries in various alloy/heat treatment

combinations. Other approaches include the effect of interactions between lattice and grain boundary dislocations [30], including dislocation "spreading" in the boundary (e.g. 31) intergranular embrittlement (e.g. 30) and the concept of structural/phase transformations at the boundary leading to the production of grain boundary phase diagrams. (e.g. 32)

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Figure 7. Transmission electron micrographs exhibiting cell formation near to grain boundaries in deformed polycrystals a) aluminium after a strain of 0.1 (courtesy of I.Barker and J. Preston). b) nickel after a strain of 0.8 and a heat treatment which has lead to a partially recovered (here) and partially recrystallised substructure.

